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PERCHLORIC ACID HOOD SAFETY II: WASH-DOWN SYSTEM DESIGN AND TESTING .

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Use of perchloric acid at or near its boiling point ("fuming") produces acid vapors and mist which can condense and accumulate in laboratory hoods, forming unstable perchloric acid crystals or perchlorates. Typically, a wash-down system is installed in perchloric acid exhaust systems to allow the rinsing of the stack, fan, ducting and plenum. However, guidance on the design, operation and testing of wash-down systems is scattered, anecdotal and often contradictory. Experience at the Lawrence Livermore National Laboratory (LLNL) suggests that standard industry wash-down designs may not be adequate to prevent explosions. Further, some newer designs are particularly difficult to wash-down. A review was conducted to determine the most effective design and operation parameters of a perchloric acid hood wash-down system.

Most perchloric acid exhaust systems, including systems currently marketed for this purpose, provide only a single valve to turn on the entire wash-down system. In 1990, an explosion occurred in a perchloric acid exhaust system at LLNL. It was ultimately determined that the explosion occurred because the top (stack) wash-down nozzle had failed to activate during wash-down, leaving the stack and fan unwashed. All perchloric acid systems in use at LLNL now incorporate 2 or more separate wash-down valves, which allow the user to confirm that all of the nozzles are working.

No definitive guidance is available regarding the duration of the wash-down cycle or amount of water required. Recently, a new perchloric acid exhaust system was installed at LLNL, with a centrifugal rather than the more traditional vane-axial fan. The manufacturer's recommended wash-down time was 10-15 minutes, but they could provide no rationale or empirical basis for this recommendation. In our experience, this was excessive and would needlessly contribute to the accumulation of potentially hazardous wash-down waste. We sought to establish the duration of the wash-down cycle empirically.

Sequential sampling of the rinsate from the new hood was conducted and subjected to analysis by the methylene blue method (qualitative) and with a perchlorate ion selective electrode (quantitative). The results indicated that effective wash-down required 4-5 minutes and about 8 gallons of water. Similar testing on a perchloric acid exhaust system with a vane-axial fan indicated that effective washing was achieved in only one minute.

It was concluded that: 1) Each wash-down nozzle or set of nozzles should be equipped with a separate valve; 2) One manufacturers wash-down recommendation of 10-15 minutes is excessive; 3) It is much more difficult to wash down an exhaust system which incorporates a centrifugal fan than a system with a vane-axial fan; 4) The methylene blue field test for perchlorates in marginally adequate, and; 5) The perchlorate ion selective electrode was a quick, accurate and sensitive way to test perchloric acid exhaust system rinsate.Finally, a theoretical model was developed which allows specific defects in washdown performance to be determined by plotting the concentration of perchlorate in the rinsate against time.

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